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Flow Testing of Cytec FM300 and FM300-2K Structural Adhesives

Roger Vodicka

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Airframes and Engines Division
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ABSTRACT

The flow of Cytec FM300 and FM300-2K structural film adhesives was examined using flow tests employing an autoclave or by using dead-weights to apply pressure. The effect of ageing the adhesive at room-temperature was examined for periods of up to 60 days. FM300 adhesive was found to show minimal changes in flow and it was difficult to determine the adhesive age from the flow test results. The effects of ageing on FM300 are most likely to appear in the results of mechanical tests such as the short overlap shear test. FM300-2K showed a more consistent change in flow with age. The flow of FM300-2K changed markedly over the ageing period (about 30-40% change in area flow) and decreased in an almost linear fashion with age. This makes it viable to determine the effects of ageing on FM300-2K based on the results of flow tests. Both types of pressure application (autoclave and dead-weights) are suitable for flow testing and offer key advantages and disadvantages. If an autoclave is available it is preferable to perform flow tests at 100 kPa.

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Flow Testing of Cytec FM300 and FM300-2K Structural Adhesives

Executive Summary

Epoxy film adhesives are used throughout the F/A-18 aircraft for adhesive bonding of honeycomb sandwich structures and other components. These film adhesives give high temperature properties and are typically cured at 120°C or 177°C.

Two commonly used film adhesives are Cytec FM300 and FM300-2. Cytec FM300 was used in the original manufacture of most of the bonded F/A-18 components including the rudder and horizontal stabilator. FM300-2 is now used as a lower-temperature curing alternative to FM300 for certain repair operations.

The storage of film adhesives at -18°C is required, as any exposure to elevated temperatures will advance the cure state of the adhesive. An advanced state of cure makes the adhesive difficult to process as it will be less inclined to flow and 'wet out' the bonding surfaces. This can cause reductions in mechanical properties such as shear strength. Material which has been stored for some time may need to be tested to ensure that it is still within life. The use of adhesive flow tests can form part of a suitable acceptance criteria for film adhesives. A flow test examines the amount of adhesive flow which occurs for a given processing pressure under a given temperature cure cycle.

This work examines the different types of flow tests that are available and examines the changes in flow of Cytec FM300 and FM300-2K when exposed to periods of room temperature for up to 60 days. FM300 adhesive was found to show minimal changes in flow and it was difficult to determine the adhesive age from the flow test results. The effects of ageing FM300 for up to sixty days on mechanical properties would need to be determined before the material could be re-qualified for use. FM300-2K showed a more consistent change in flow with age making it viable to determine the effects of ageing on FM300-2K based on the results of flow tests.

The costs and logistics of storing film adhesives for use in repair operations are high and significant cost savings are possible if the film adhesives can be effectively used beyond the manufacturer's stated shelf life. This report has identified some important issues associated with the re-lifing of two of the main film adhesives used by the RAAF.

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Roger Vodicka graduated BSc. (Hons.) from Monash University and joined AMRL in 1990. He has previously worked on a range of projects at AMRL including adhesively bonded composite repair technology, battle damage repair methods, effects of environment on the durability of composite materials as well as the durability of honeycomb sandwich structures. He is currently the task manager for the Composite Repair and Engineering Development Program (CREDP).

Contents

1. Introduction

Structural film adhesives are used extensively in the aerospace industry for adhesive bonding of both metallic and composite materials. They are convenient and practical to use since they allow the adhesive to be used in the form of a sheet rather than a viscous liquid allowing easier manufacture and control of the amount of material used. Epoxy film adhesives are used throughout the F/A-18 aircraft for adhesive bonding of honeycomb sandwich structures, metal to metal bonding and metal to composite bonding. These film adhesives give high temperature properties and are typically cured at 120°C or 177°C.

Two commonly used film adhesives are Cytec FM300 and FM300-2K. Cytec FM300 was used in the original manufacture of most of the bonded F/A-18 components including the rudder and horizontal stabilator. Cytec FM300-2K is a lower-temperature curing adhesive and has been approved as a substitute for FM300 when used for structural repairs to some honeycomb components, providing revised repair guidelines are used (ie: the Boeing Bonded Joint Analysis Methodology BJAM). The lower temperature cure of FM300-2K (120°C) is an advantage during repair of honeycomb sandwich structure as it reduces the risk of boiling any water that may be present within the honeycomb. High pressures can arise from steam at high temperatures and this can cause the failure of a component due to the bonded skin separating from the honeycomb core or the core itself can be damaged.

The storage of film adhesives at -18°C is required, as any exposure to temperature will advance the cure state of the adhesive. The epoxy adhesive undergoes an irreversible chemical reaction (ie: curing). An advanced state of cure makes the adhesive difficult to process, as it will be less inclined to flow and 'wet out' the bonding surfaces. This can also cause reductions in strength properties such as shear strength. Cytec FM300-2K will remain in useful life for up to one year at -18°C according to manufacturer's recommendations. FM300 has a shelf life of 6 months at -18°C. If the temperature of storage increases, the effective 'life' of the material will be reduced. FM300 can remain in life for up to 30 days when stored at 21°C. FM300-2K can remain in life for up to 10 days at 24°C. It is therefore vital to ensure that any adhesive used for aircraft repair and manufacture is within shelf life.

Material that has been stored for some time may need to be tested to ensure that it is suitable for use on aircraft. Material which is outside of the manufacturers stated shelf life can be re-qualified for further use provided certain acceptance tests are passed. Such testing may include the use of flow tests or mechanical tests such as the short overlap shear strength test. RAAF Standard (ENG) A5007 describes the standards that should be used when film adhesives are received, stored and used. The use of adhesive flow tests is listed as one of the acceptance tests required to re-qualify film adhesives. A flow test examines the amount of adhesive flow which occurs for a given processing pressure under a given temperature cure cycle.

This work examines the different types of flow tests that are available and examines the changes in flow of Cytec FM300 and FM300-2K when exposed to room temperature for periods of up to 60 days.

2. Materials Used

Two types of epoxy film adhesive used for F/A-18 manufacture and repair are examined in this study. Both are manufactured by Cytec-Fiberite in the U.S.A. The material batches used in the study are shown in Table 1.

Table 1 Details of film adhesives used for flow testing

Name	Areal Weight (lbs/ft^2)	Date of Manufacture	Product # - Batch - Roll/Can/Sheet
FM3002-K	0.10	25th January 1999	4169-00531-00004
FM300	0.10	20th July 1999	4279-01042-00009B

2.1 Adhesive Cure and Processing

The cure cycles for both adhesive materials employ a ramp to the cure temperature at 3°C per minute followed by a dwell at the cure temperature. In all cases in this study it was only necessary to cure the adhesive to the gel stage (ie: where no further flow occurs). FM300 was cured for 30 minutes at 177°C while FM300-2K was cured at 120°C for 30 minutes.

The cure pressure is another important aspect of adhesive processing. It ensures that the adhesive flows and is compacted to the desired bondline thickness. Bondline thickness is often controlled using a scrim cloth. A scrim cloth is typically a woven nylon fabric weave placed within the film adhesive at time of adhesive manufacture. The 'K' suffix denotes a knit weave for the scrim fabric. A mat scrim cloth is denoted 'M'. Adhesives can also be supplied without a scrim cloth (unsupported) and have a 'U' suffix. The scrim cloth will affect the result of any flow test so it is vital to ensure that flow tests are compared for the same resin system, identical scrim cloth as well as the same test procedure and cure conditions. Pressure can be applied in a number of ways depending on the process. This includes using an autoclave, mechanical press or the use of dead weights. The last method is unlikely to be used in the manufacture or repair of aerospace parts.

3. Adhesive Ageing

Both adhesive systems were aged by exposing them in the laboratory environment for periods of between zero and sixty days. A baseline test using fresh material represented zero days of exposure. Laboratory air temperature was approximately

21°C. Adhesive discs were aged inside sealed aluminium coated plastic bags to prevent contamination and moisture uptake.

A period of sixty days was chosen since this is the maximum amount of time that the adhesives are likely to be re-qualified. Some practices allow a material to be re-qualified for another 50% of its original shelf life. For FM300 this represents the material being re-qualified more than twice. For FM300-2K the sixty day period represents a large period in excess of its original shelf life (ie: ten days).

4. Flow Test Techniques

The flow test technique used here is based on the Boeing Support Standard BSS 7240 'Adhesive Flow Test' that is recommended by CytecFiberite [1]. The standard requires the use of a 1.5" (38 mm) diameter disc of material placed between mylar separator film which is then cured according to the manufacturer's recommendation. The flow was then measured by calculating the increase in adhesive area. This was done by comparing the adhesive flow with a simple series of concentric circles that mark the relevant flow levels.

The technique used in this study is a slightly modified version of the Boeing standard (See Appendix A). Teflon coated fibreglass was used as the separator film and a 32mm diameter adhesive sample was cut out for the test using a hole punch. Care was taken to ensure that the adhesive was cut cleanly. Samples were placed under pressure using either an autoclave or by placing dead-weights on the material discs. They were then cured using the cure cycles defined in Section 2.1.

4.1 Autoclave Based Flow Test

Two autoclave pressures were used, 100 kPa and 280 kPa (280 kPa is recommended by the manufacturer). It was found that 6 discs of material could easily be cured on a single aluminium plate. This made it easy to perform a number of replicates at the same time. The test setup is illustrated in Figure 1. The whole setup was covered in vacuum bagging material before processing as per standard autoclave curing practice.

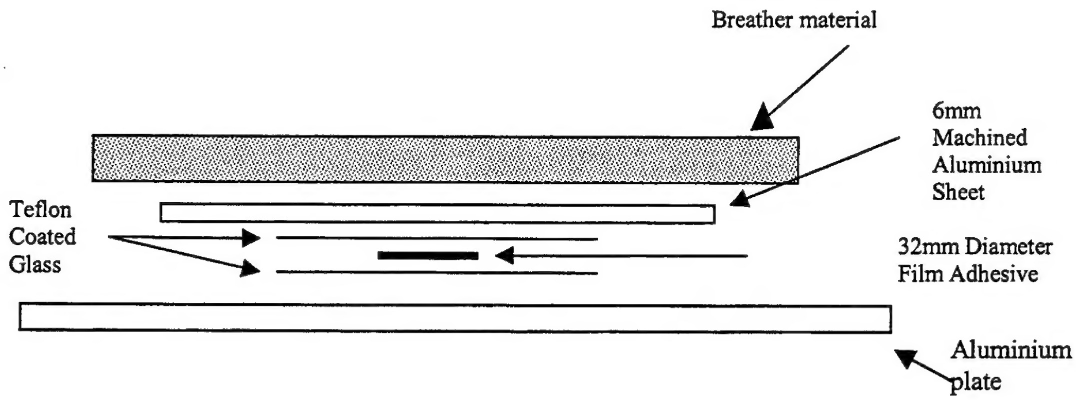


Figure 1 Test setup for autoclave-based adhesive flow test.

4.2 Dead weight flow method

Another technique was also trialed which did not require the use of an autoclave or a mechanical press. In this technique pressure to the disc of adhesive was applied using a dead weight. Heat was then applied as per the manufacturer's recommend cure cycle using a heater blanket. Heater blankets are typically used by the RAAF for adhesively bonded repairs and are generally more available and less expensive than an autoclave or mechanical press. As per the method above, a 32mm diameter disc of material was used and placed between teflon coated fibreglass. In this study two dead weights were used to simulate pressures of 100 kPa(8.35 kg dead weight) and 280 kPa (22.27 kg dead weight). The test setup is illustrated in Figure 2.

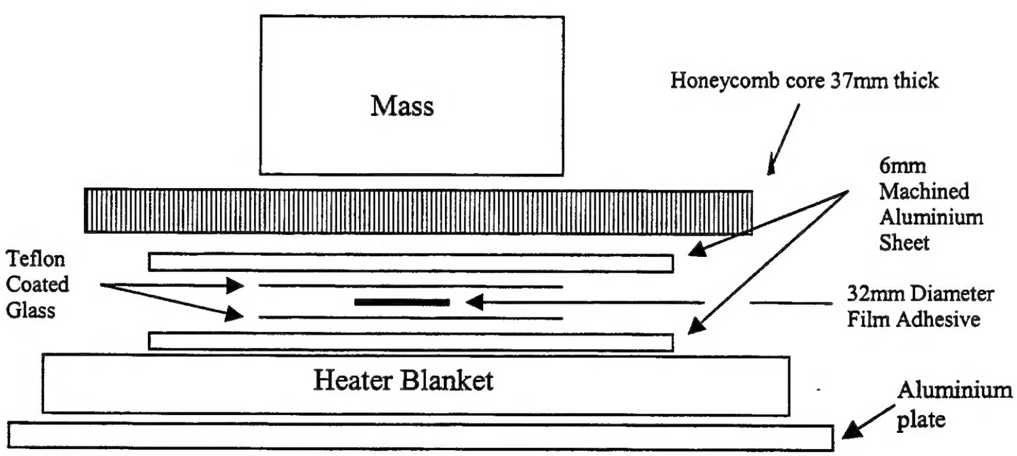


Figure 2 Test setup for dead-weight adhesive flow test

Temperature was applied using a heater blanket and controlled using a MACBONDER [2] temperature controller. A thermocouple was placed on the 6mm thick aluminium sheet directly above the adhesive disc. A piece of honeycomb core was used to thermally insulate the large dead weight from the test area

4.3 Measuring Flow

There are a number of ways to measure the resulting flow after the adhesive has been processed. These include flow change by weight and change by area.

4.3.1 Flow Change by Weight

In this measurement method the sample disc is weighed prior to testing on a four-figure balance. After processing the cured disc is cut using the same size hole punch as for the original disc (ie: 32mm) and this cut-out is then weighed. The cut-out section must include the entire scrim cloth. The flow is then a percentage of the original weight

ie: $\%\,\text{flow} = (1 - \dfrac{\text{Mass of flow cutout}}{\text{original mass}}) * 100$.

4.3.2 Flow by Change of Area

The change in area after the material is cured can be calculated using a number of methods. The Boeing Support Standard BSS 7240 'Adhesive Flow Test' uses a series of concentric circles over which the adhesive disc is placed. The circles then indicate the percentage flow.

Another way is to measure the average diameter of the disc before and after processing and express the flow as: $\dfrac{\text{Final Flow Area}}{\text{Original Area}} * 100$.

In this study an average flow area was calculated by measuring the flow disc diameter at four points. In most cases the flow disc was roughly circular and a good estimate of area could be gained using this technique.

5. Results

Results are shown for both processing techniques (autoclave and dead weight) and for both methods of calculating the flow (weight and area). The results are plotted against the ageing period of the adhesive at room temperature. Tests were conducted during the period December 1999 and March 2000.

5.1 FM300 Autoclave Cure

Figure 3 shows the flow test results for cure of FM300 in the autoclave at 100 kPa. Measurements of both area and weight were taken and are plotted on alternate y-axes.

The change in flow over the ageing period is very low and there are some large error bars which make it difficult to assess any changes due to ageing.

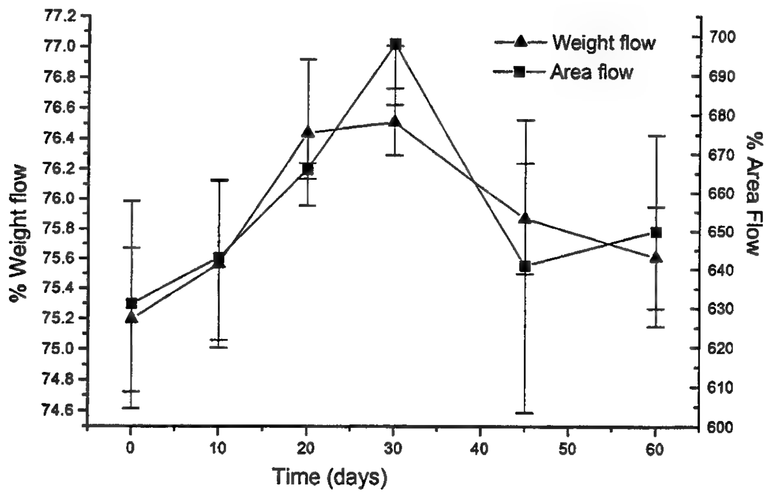
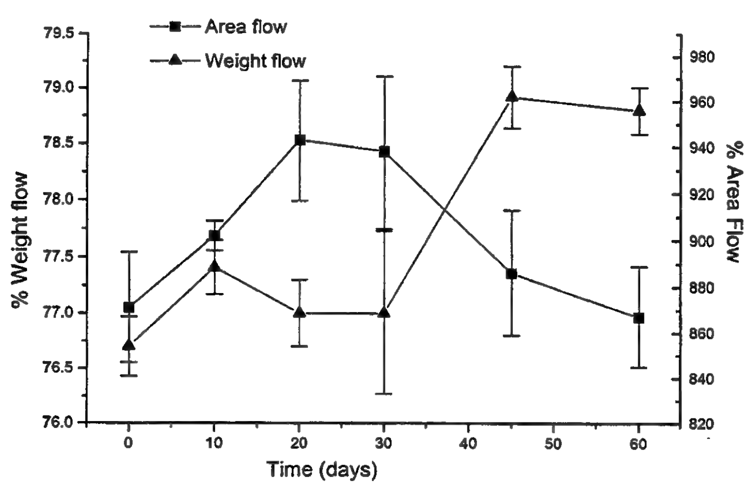


Figure 3 Flow test result for 100 kPa autoclave cure of FM300.

Figure 4 shows the flow test results for autoclave cure at 280 kPa. The flow by weight sees an increase after 30 days while the flow area peaks around the 20 to 30 day times. There is also less correlation between the weight and flow area measurement when compared to all the other results. Again the change in flow is very small over the ageing period and the sizes of the error bars make it difficult to assess actual flow changes.



.Figure 4 Flow test result for 280 kPa autoclave cure of FM300.

5.2 FM300 Dead Weight Cure

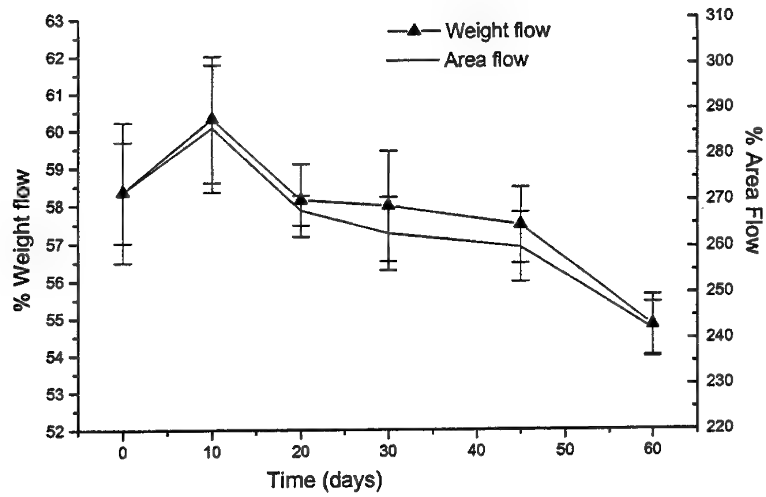


Figure 5 Flow test result for 100 kPa dead-weight cure of FM300.

Figure 5 shows the flow test result for the dead-weight cure of FM300 at 100 kPa. The flow sees an initial rise after 10 days followed by a marked decrease after 60 days. The level of flow is also much less than that seen when using an autoclave for the same given pressure.

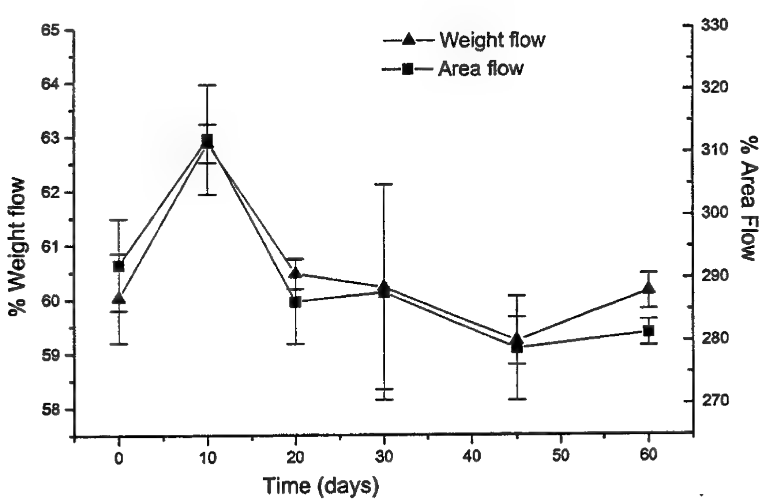


Figure 6 Flow test result for 280kPa dead-weight cure of FM300.

Figure 6 shows the flow test result for the dead-weight cure of FM300 at 280kPa. The results show an increase in flow after 10 days. After 20 days the flow is back to the same level as zero days of ageing. Small decreases are then noted when ageing continued to 60 days. The change in flow over the ageing period is small and the size of the error bars makes it difficult to detect the age of the material by its flow.

5.3 Autoclave Cure FM300-2K

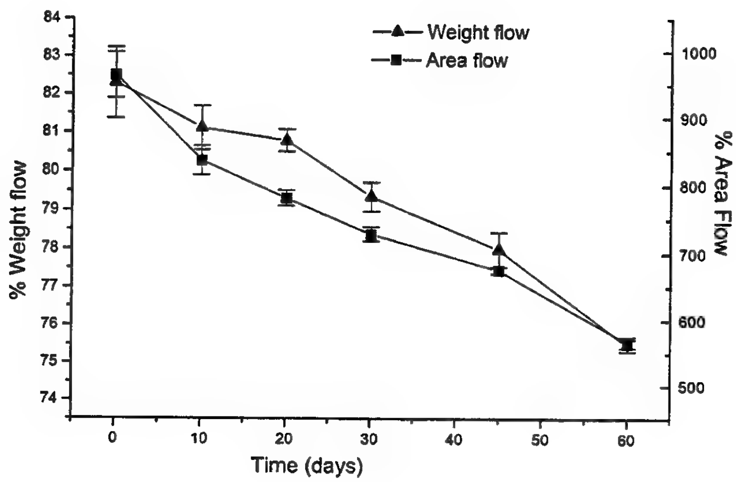


Figure 7 Flow test result for 10 0kPa autoclave cure of FM300-2K

Figure 7 shows the results of flow tests for the 100kPa autoclave cure of FM300-2K. The flow sees a steady, almost linear, decline in flow over the entire ageing period. The magnitude of the change in flow is significantly greater over the ageing period compared to FM300.

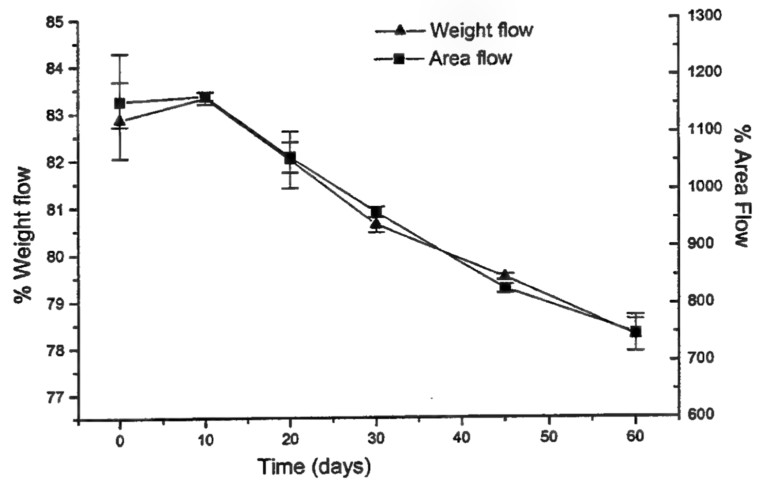


Figure 8 Flow test result for 280 kPa autoclave cure of FM300-2K

Figure 8 shows the results of flow tests for FM300-2K cured in the autoclave at 280 kPa. For the first 10 days the flow is essentially unchanged. After this the flow sees a linear decline in flow up to the 60 days of ageing investigated.

5.4 FM300-2K Dead Weight Cure

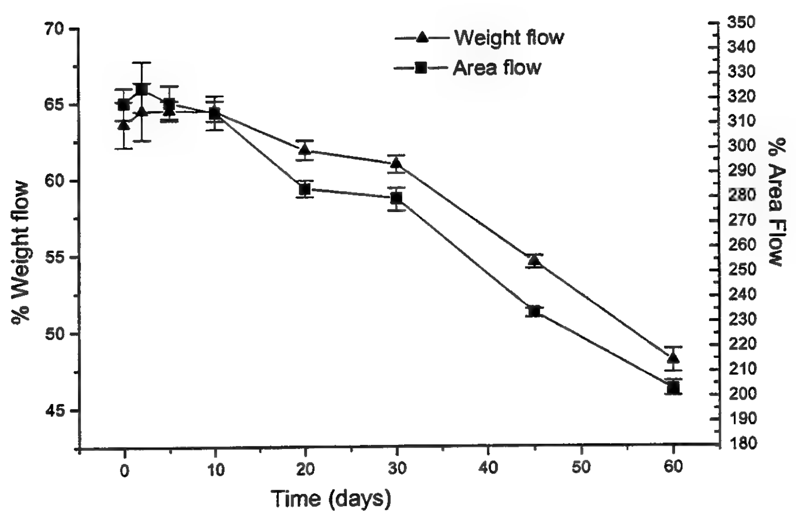


Figure 9 Flow test result for 100 kPa dead-weight cure of FM300-2K

Figure 9 shows the results of flow tests for the 100 kPa dead-weight cure of FM300-2K. The flow is unchanged after 10 days and then sees a linear decline until 60 days.

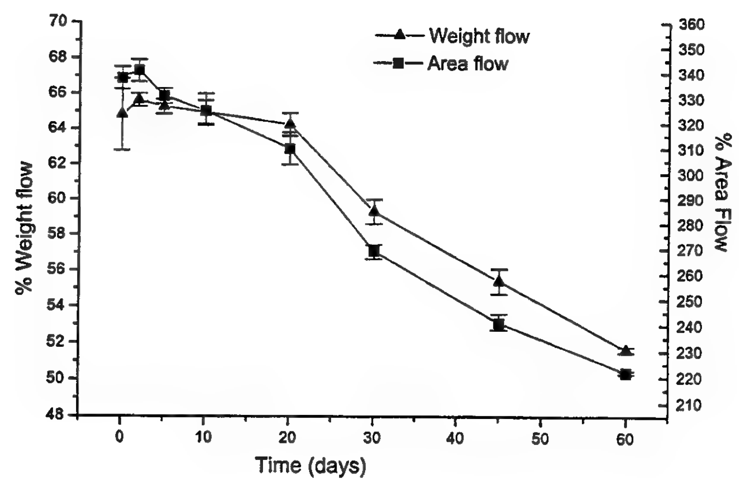


Figure 10 Flow test result for 280kPa dead-weight cure of FM300-2K.

Figure 10 shows the results of flow tests for the dead-weight cure of FM300-2K at 280 kPa. The flow is unchanged after five days but then sees a linear decline for ageing periods up to the 60 days investigated.

6. Discussion

The results shows that the difference in measuring the flow using area or weight does not seem to have a great bearing on the outcome of the results. The area can be difficult to measure if the flow is not perfectly circular. The weight can also be difficult to measure if the cured adhesive fractures when the hole punch is used to cut the material. The exception to this was the result of curing FM300 at 280 kPa in an autoclave. It is not known why these results are inconsistent.

The results for FM300 suggest that the flow can increase for ageing up to thirty days after which it declines back to original (un-aged) levels. The amount of change in flow due to ageing is quite low in FM300. In most cases the fluctuation in flow over the ageing period is less than 10%. In many cases it would be difficult to assess the level of ageing of FM300 based on the flow test for ageing periods of up to sixty days. FM300 cures at a higher temperature compared to FM300-2K and is thus less likely to be affected by room temperature exposure. This is borne out in the fact that FM300 may be stored for thirty days at 21°C while FM300-2K can be stored only ten days at 24°C. Only the 100 kPa dead-weight method shows a clear decline in flow over the ageing period. This tends to indicate that the flow of FM300 is likely to remain satisfactory even after extended ageing. The mechanical properties of FM300 after ageing are another issue. A good flow test cannot guarantee good lap shear strength or other adhesive based mechanical properties. From this perspective the use of short overlap

shear strength tests as suggested in RAAF STD (ENG) A5007 may be preferable for the re-qualification of FM300.

The change in flow for FM300-2K was much easier to detect and undergoes a larger change in magnitude over the ageing period. The flow of FM300-2K is essentially unchanged after 10 days in most test cases but then undergoes an approximately linear decline in flow. Over the sixty day ageing period the flow changes by about 30 to 40% (area flow). This is much greater than for FM300. It would be a relatively easy task to correlate ageing with flow for FM300-2K using any of the methods discussed. This makes the flow testing of FM300-2K a useful way of assessing the age of the material.

Table 2 shows a comparison of the two flow test methods examined in this study. The results show that the dead-weight method produces generally less flow than that of the autoclave. For this reason the flow testing of FM300-2K using the autoclave is preferable if the equipment is available. The use of 100kPa pressure seems to show the effects of ageing better than 280kPa especially in the early ageing period (around ten days).

Use of the dead-weight method is a viable alternative. It must be noted that the technique can be unreliable if the weight is not carefully assembled over the adhesive disc. For this reason it is advised that at least three concordant data points be used to define the flow of FM300-2K adhesive.

Table 2 Advantages and disadvantages of differing flow test methods

Method	Advantages	Disadvantages
Dead-weight	a) Equipment readily available b) Low cost	a) Can only reasonably test single adhesive disc at one time b) Accuracy relies on correct placement of weight on disc
Autoclave	a) Can cure up to six discs at once b) Pressure is evenly applied	a) High cost of autoclave purchase b) High running costs c) Time taken to set up vacuum bag, breather and curing plate before processing.

7. Conclusions

The flow of Cytec FM300 and FM300-2K epoxy film adhesives was examined using flow tests employing an autoclave or by using dead weights to apply pressure. The effect of ageing the adhesive at room temperature was examined for periods of up to 60 days. FM300 adhesive was found to show minimal changes in flow and it was difficult to determine the adhesive age from the flow test results. The effects of ageing on FM300 will most likely need to be determined using a mechanical test such as a short overlap shear strength test. FM300-2K showed a more consistent change in flow with age. The flow of FM300-2K changed markedly over the ageing period (about 30-40% change in area flow) and decreased in an almost linear fashion with age. This makes it viable to determine the ageing effects on FM300-2K based on the results of flow tests. Both types of pressure application (autoclave and dead-weights) are suitable for flow testing and offer key advantages and disadvantages. If an autoclave is available it is preferable to perform flow tests at 100kPa.

8. Recommendations

1. For both adhesives the flow test results need to be correlated with mechanical properties to ensure that the adhesive is suitable for the end application. A flow test level criteria may then be set based on the changes in mechanical properties versus time of exposure. For FM300-2K this criteria could be formed using the results of mechanical tests and the flow test data in this paper.
2. The lack of flow change in FM300 would require further investigation to assess whether any changes in associated mechanical properties are observed. It would be an important outcome if the shelf life of FM300 could be significantly extended. This would dramatically reduce material costs. A suitable acceptance test would then need to be devised for the re-lifing of the adhesive for these extended shelf lives.
3. Consideration needs to be given to extending this work to include Cytec FM73 adhesive which is widely used by the RAAF for bonded composite repairs.

9. Acknowledgments

The author would like to acknowledge the technical efforts of Mr. Peter Haggart for meticulously performing all the flow testing detailed in this study.

10. References

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1 Fax communication from CytecFiberite from Christopher DiGiacomo, 3rd January 1999.

2 MACBONDER temperature control system, DSTO-AMRL. Produced by Dr Alan Wilson.

Appendix A: Flow test preparation procedure

a. Remove FM 300-2K (or FM300) film adhesive from freezer and allow to thaw to room temperature before opening the bag.

b. Remove film adhesive from bag and punch out one sample of 32 mm diameter adhesive using a hole punch.

c. Remove backing film from adhesive and weigh the circular adhesive piece using a 4 digit balance.

d. Cut two square pieces of non-porous teflon coated glass (Airtech International (USA) "Release-Ease 234TFNP") with an approximate side length of 150 mm.

e. Prepare the test setup as shown in Figure 1 (Dead-weight) or Figure 2 (Autoclave).

f. Commence heating the adhesive and sandwich assembly at a rate of 3°C/ minute up to the cure temperature. This is 177°C for FM300 and 120°C for FM300-2K.

g. Dwell at cure temperature for 30 minutes.

h. Remove adhesive flow sample from between the teflon coated glass.

i. Measure diameter of the flow disc at four points and calculate area.

j. Punch out a section from the centre of the adhesive flow sample using a 32 mm diameter hole punch being careful to include the entire scrim cloth.

k. Mass this 32 mm adhesive flow sample and calculate weight flow.

The report shall include the following:

a. Complete identification of the adhesive being tested including type, weight, batch number, roll number and date of manufacture. Include all prior thermal history of material, if known ie: freezer life and room temperature life remaining.

b. Date of test.

c. Name of person conducting the test.

d. Description of test method. Include details of insulator material used as well as type of teflon used for flow.

e. Condition of adhesive after curing. For example, heavily voided or discoloured.

f. Result of each test.

g. Average of three test results per each test condition expressed in percentage flow (area or weight).

h. Any observations or notes taken during testing.

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No Limitations

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18. DEFTEST DESCRIPTORS

Epoxy resins; Adhesive bonding; Sandwich structures; Honeycomb structures; Composite materials; F/A-18 aircraft; Stabilators; Rudders; Mechanical tests; Shear tests

19. ABSTRACT

The flow of Cytec FM300 and FM300-2K structural film adhesives was examined using flow tests employing an autoclave or by using dead-weights to apply pressure. The effect of ageing the adhesive at room-temperature was examined for periods of up to 60 days. FM300 adhesive was found to show minimal changes in flow and it was difficult to determine the adhesive age from the flow test results. The effects of ageing on FM300 are most likely to appear in the results of mechanical tests such as the short overlap shear test. FM300-2K showed a more consistent change in flow with age. The flow of FM300-2K changed markedly over the ageing period (about 30-40% change in area flow) and decreased in an almost linear fashion with age. This makes it viable to determine the effects of ageing on FM300-2K based on the results of flow tests. Both types of pressure application (autoclave and dead-weights) are suitable for flow testing and offer key advantages and disadvantages. If an autoclave is available it is preferable to perform flow tests at 100 kPa.

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